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COMBINED  $\text{He}^3$  CRYOSTATS  
AND  $\text{He}^3\text{-He}^4$  DILUTION REFRIGERATORS

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COMBINED  $\text{He}^3$  CRYOSTATS AND  $\text{He}^3\text{-He}^4$  DILUTION REFRIGERATORS

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## ABSTRACT

Equipment built for low temperature range 0.05 - 1.5 K solid state physics research, at the Central Research Institute for Physics in Budapest is described. The  $\text{He}^4$  Dewar and the connected systems are able to accommodate two different interchangeable types of inserts, should there be a need to switch from a  $\text{He}^3$  evaporation system to a dilution refrigerator or vice versa.

## РЕЗЮМЕ

Производится описание созданной в ЦИФИ комбинированной установки, которая представляет собой  $\text{He}^3$  испаритель и систему с раствором  $\text{He}^3\text{-He}^4$ . Установка может быть применима для низкотемпературных исследований физики твердого тела в диапазоне 0,05 К - 1,5 К, располагает двумя различными охлаждаемыми элементами, которые могут быть заменены в зависимости от того, что в данном случае требуется ли эксплуатация  $\text{He}^3$  испарителя, или же системы с раствором  $\text{He}^3\text{-He}^4$ .

## KIVONAT

Egy kombinált  $\text{He}^3$  elpárologtató vagy  $\text{He}^3\text{-He}^4$  oldásos hűtőberendezést ismertetünk, amely a KFKI-ban készült. Nagyon alacsony hőmérsékletű szilárdtestfizikai kutatásokra 0,05 K - 1,5 K tartományban alkalmazható, két különböző hűtőbetéttel, amelyek cserélhetők, attól függően, hogy  $\text{He}^3$  elpárologtató vagy  $\text{He}^3\text{-He}^4$  oldásos rendszert kívánunk-e működtetni.



## INTRODUCTION

Temperatures down to 1 K can be reached by pumping on the  $\text{He}^4$  bath with large rotary pumps; lower temperatures are difficult to obtain due to the very low vapour pressure of  $\text{He}^4$  and the presence of the  $\lambda$  transition and the resulting superfluid.

The helium isotope of mass 3 has a considerably higher vapour pressure as can be seen in Fig. 1, with no  $\lambda$  transition in the discussed region so by pumping on the liquid  $\text{He}^3$  it is fairly easy to reach temperatures as low as 0.3 K [1,2,3].

The cooling power of this type of cryostat decreases exponentially with temperature making tasks at lower temperatures extremely difficult.

One solution is to apply another process: that of dilution refrigeration. The dilution of  $\text{He}^3$  in  $\text{He}^4$  enables temperatures as low as 10 mK to be maintained continuously even with a relatively large heat leak. However, devices capable of this are rather complicated [7]. Simple dilution refrigerators can easily reach 50 mK with large heat leaks and give a cooling power over 10  $\mu\text{W}$  at 100 mK [8].

Cooling in the dilution refrigerator is produced by causing the  $\text{He}^3$  atoms from the upper almost pure  $\text{He}^3$  - concentrated phase, to move across the phase separation boundary into the lower dilute, superfluid phase. For detailed principles of dilution refrigeration see Ref. [4-7].

It is apparent /Fig. 2/ that the greatest cooling power from 1.5 K to 0.4 K can be reached with a  $\text{He}^3$  cryostat lower down the scale by a dilution refrigerator. Both the  $\text{He}^3$  cryostat and dilution refrigerator in Fig. 2 are pumped with a system able to handle  $\sim 30$  lit/sec of gas in the appropriate pressure range.

For this reason we have designed a modular set of equipment which consists of a  $\text{He}^4$  Dewar vessel with a very low evaporation rate, having a pumped  $\text{He}^4$  bath in which either a  $\text{He}^3$  cryostat or a dilution refrigerator within removable inserts can be placed. Any of them can be simply and rapidly connected to the versatile Dewar, auxiliary  $\text{He}^4$ -, vacuum; and  $\text{He}^3$  and  $\text{He}^3$ - $\text{He}^4$  systems.



Two such sets have already been completed and can be used at temperatures from 1.5 K to 0.05 K for thermodynamic and neutron diffraction measurements. They have been constructed on the basis of the experience gained at building a  $\text{He}^3$  cryostat [3] and a dilution refrigerator at the P.J. Safarik University, Košice and a dilution refrigerator in Budapest [9].

## A. DESCRIPTION OF THE DESIGN

The equipment is based on a  $\text{He}^4$  Dewar vessel with a pumped helium bath. Into this bath kept at  $\sim 1.2$  K is immersed the chosen insert with either a  $\text{He}^3$  cryostat or a dilution refrigerator built into the vacuum jacket suspended on the pumping tubes. The Dewar as well as the chosen insert are connected to the same versatile auxiliary room temperature systems described in section A 3.

### A. 1. CONSTRUCTION OF THE $\text{He}^3$ CRYOSTAT INSERT

The inner parts of the  $\text{He}^3$  insert are shown in Figure 3.

$\text{He}^3$  gas is led in and condensed in tube I and is pumped from tube II. The small diameter tube I can be used during the  $\text{He}^3$  pumping for  $\text{He}^3$  vapour pressure measurements and thus for temperature determination /Fig. 5/ /M6 and M7/. Pumping line III is connected to a high vacuum system to control the pressure in vacuum jacket provided with an exchange gas inlet. The wires to thermometers and heaters are also led through this tube if necessary. Both the  $\text{He}^3$  and the vacuum pumping lines are furnished with light radiation traps; the pumping lines are made of thin-walled stainless steel tubes and expand in diameter with rising temperature; the  $\text{He}^3$  pumping line from 12 mm in liquid He to 32 mm at the top of the Dewar; the vacuum line from 8 mm to 20 mm.

The vacuum jacket /6/ is removable, sealed with an indium "O"-ring. The condenser /1/ made of the inlet CuNi tube, 2.5 mm i.d. in form of spiral, is over the upper flange of the jacket.

The evaporation chamber /3/ is made of copper; the bottom is machined so as to enlarge the surface in contact with liquid  $\text{He}^3$ . The samples can be connected into one threaded boring M10 and four others M4 in the lower end of the chamber. In this cold finger /5/ are the grooves providing the thermal anchoring of wires led to thermometers and heaters and a boring for the thermometer /4/.



Cryostat No I is provided with a  $12.5 \text{ cm}^3 \text{ He}^3$  evaporation chamber cryostat No II with one of  $15 \text{ cm}^3$ .

## A.2. CONSTRUCTION OF THE DILUTION REFRIGERATOR INSERT

The inner parts of this are shown in Fig. 4.

It is of a simple design placed in a removable vacuum jacket /11/ sealed with indium. On the upper side of the mounting flange are the pumping ports II and III and a condenser /1/ made of a 0.8 mm i.d. 20 cm long copper tube, wound in a spiral, connected to the  $\text{He}^3$  input line made of a 1 mm i.d. thin-walled CuNi tube. A flow impedance /2/ is located at the output of the condenser, under the flange. Its value was set to  $6 \times 10^{-3} \text{ cm}^3 \text{ sec}^{-1}$  STP  $\text{N}_2$  gas flow at room temperature and a pressure drop of  $\sim 200$  torr.

The still /3/ is copper; the orifice in its top, to reduce the  $\text{He}^4$  film flow is 1 mm in diameter. The still has a volume  $9 \text{ cm}^3$  in No I and  $12 \text{ cm}^3$  in dilution refrigerator No II.

No. II is constructed to enable step-heat exchangers to be accommodated but they have not as yet been fitted.

Within the still is the still exchanger /4/ made of a copper 0.8 mm i.d. tube 100 cm long. The heater /5/  $\sim 600$  ohms is wound on the outer surface of the bottom of the still, where a thermometer /9/ is also placed.

The coaxial counterflow heat exchanger /6/ is made of two thin-walled CuNi tubes, the inner one having a 0.25 mm i.d., the outer one 1 mm i.d. They are both bent into a spiral together. The heat exchanger for No. I is 120 cm long; that for No II, 90 cm long. There is a copper strip thermal ground at the colder 1/3 of the spiral.

The  $4 \text{ cm}^3$  volume copper mixing chamber /7/ is fixed on a very thin walled stainless steel support tube. The lower bottom of the mixing chamber is machined on the inside so as to enlarge the surface in contact with liquid. The cold finger /8/ is of the same design as those on the  $\text{He}^3$  cryostats /Fig.3/. There are also the grooves for thermal anchoring and the thermometer /10/.

The  $\text{He}^3$  from the still is pumped off through II and the vacuum in the jacket is controlled and monitored via III.

The pumping lines II and III are provided with light radiation traps and are made of thin walled stainless steel tubes widening in diameter. For the  $\text{He}^3$  pumping line the diameter expands from 12 mm at He temperatures to



45 mm at the top of the Dewar; for the vacuum line from 8 mm to 20 mm. The thermometer and heater wires also are connected via III. This has the advantage of room temperature vacuum tight lead-throughs.

### A.3. THE ROOM TEMPERATURE SYSTEMS

The Dewar vessel with the chosen insert with fast dismountable joints is connected to auxiliary room temperature laboratory vacuum and plumbing systems shown in Fig. 5 which consist of

- a/  $\text{He}^4$  pumping and recovery system,
- b/ high vacuum system - to maintain and monitor the isolation vacuum in the vacuum jacket. There is a valve for letting exchange gas into the vacuum jacket if necessary,
- c/  $\text{He}^3$  and  $\text{He}^4$  mixture pumping, circulation, storage and handling system. In this the  $\text{He}^3$  and gas mixture storage tanks are of 15 l volume each. The room temperature pumping lines are 50 mm i.d. The pumping or circulation is facilitated by an Edward's Mercury Diffusion pump 35 l/sec; its pumping rate with a liquid  $\text{N}_2$  trap is lower, roughly 30 l/sec; and an Alcatel's sealed rotary pump 2  $\text{m}^3$ /hour.

It is to be noted here that if it appears necessary to switch repeatedly the operation from  $\text{He}^3$  system to dilution refrigeration and vice versa, we recommend the provision of an additional storage tank, to save the  $\text{He}^3$  trapped in rotary pump HEP 1 /Fig. 5/.

### B. PERFORMANCE

#### B.1. $\text{He}^3$ CRYOSTATS

Two inserts belonging to two different sets were examined. The cooling down diagram for both is the same and is shown in Fig. 6 compared with that of the dilution refrigerator. The downward part from point 3 of the  $\text{He}^3$  cryostat cooling down curve shows two parts; the upper corresponding to the  $\text{He}^3$  evaporation chamber pumped with rotary pump only, the lower temperature region down to 270 mK being reached by the whole pumping capacity of the system. The cooling power is shown in Fig. 7. Comparison with the cooling power of the dilution refrigerator is in Fig. 2.

Note: the condensation can be completed in approximately 5 minutes, and any temperature within the given range, even the lowest, can be reached in less than 12 minutes. The pumping rate can be controlled by a special valve.



As the existing heat leaks are small, the charged liquid  $\text{He}^3$  and the  $\text{He}^4$  in the pumped path allow at least 8 hours operation without refill.

The lowest temperature in cryostat No. I was 290 mK, that in cryostat No. II 270 mK, due to the differing heat leaks.

Temperatures are derived from the resistance of Speer 220 Ohm 1002 Grade carbon resistors calibrated in 3 points against  $\text{He}^3$  vapour pressure and  $\text{He}^4$  vapour pressure.

An a.c. resistance bridge was used to measure the resistance of the thermometers. The bridge accuracy is 1%, the power dissipation less than  $10^{-12}$  W.

## B.2. DILUTION REFRIGERATORS

Insert No. I was examined only.

The cooling down diagram, can be seen in Fig. 6; the resulting cooling power is shown in Fig. 8. Comparison with  $\text{He}^3$  cryostats in Fig. 2 shows only the curve for the greatest cooling power available in the dilution refrigerator.

The condensation time can be reduced if the condensation is also led through the pumping line.

The lowest temperature was 50 mK with circulation  $1.7 \times 10^{-5}$  moles/sec. This corresponds to 0.21 mW still power.

The largest circulation was  $1.3 \times 10^{-4}$  moles/sec, which corresponds to 5.4 mW still power and to a full heat load supplied to the mixing chamber at 300 mK.

The temperatures are derived from the resistance of 220 ohm Speer resistors placed in borings in the cold fingers of the mixing chambers. The thermometers were calibrated against  $\text{He}^3$  and  $\text{He}^4$  vapour pressure.

## CONCLUSIONS

The performance of all inserts was stable and reliable, differences between the cryostats and runs were small - nearly all within the accuracy of temperature determination. The construction of a lot of parts is identical, allowing rapid manufacture. Assembling and repairs are simple, all parts are easily accessible.



It therefore appears that a favourable piece of laboratory equipment has been designed, manufactured and tested giving satisfactory results which are in good agreement with those published elsewhere.

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# FIGURE CAPTIONS

Fig. 1 :  $\text{He}^3$  and  $\text{He}^4$  vapour pressure vs. temperature

Fig. 2 : Cooling power vs. temperature  
 $\text{He}^3$  -  $\text{He}^4$  evaporation cryostat  
 $\text{He}^3$  -  $\text{He}^4$  dilution refrigerator

Fig. 3 : Inner parts of  $\text{He}^3$  cryostat

1 - condenser	
2 - filling tube also used for vapour pressure measurements	
3 - $\text{He}^3$ port	
4 - thermometer	I - $\text{He}^3$ inlet
5 - cold finger	II - $\text{He}^3$ pumping
6 - vacuum jacket	III - vacuum

Fig. 4 : Inner parts of dilution refrigerator

1 - condenser	I - $\text{He}^3$ inlet
2 - low impedance	II - $\text{He}^3$ pumping
3 - still	III - vacuum
4 - still exchanger	
5 - heater	
6 - continuous heat exchanger	
7 - mixing chamber	
8 - cold finger	
9 - still thermometer	
10 - mix thermometer	
11 - vacuum jacket	

Fig. 5 : Layout of the room temperature systems

K-cryostat in the  $\text{He}^4$  Dewar vessel

1-20 - valves

M1 - M7 - vacuum gauges

HVG - high vacuum gauge

LVG - low vacuum gauge

KF1, KF2 - traps with liquid  $\text{N}_2$

S - trap with sintered copper

T1, T2 - storage tanks

HEP 1 - Alcatel's 2002 M sealed rotary pump

HEP 2 - Sealed rotary pump 40  $\text{m}^3/\text{h}$

HDP - Edward's mercury diffusion pump 2M4B

Fig. 6 : Cool down times

$\text{He}^3$  -  $\text{He}^3$  evaporation cryostat

$\text{He}^3$  +  $\text{He}^4$  - dilution refrigerator

1 - start of pumping on the  $\text{He}^4$  bath

2 - start of condensation

3 - start of pumping on  $\text{He}^3$  liquid in  $\text{He}^3$  evaporation cryostats

3' - start of circulation in dilution refrigerators

Fig. 7 : Cooling power of No I and No II  $\text{He}^3$  evaporation cryostats

Fig. 8 : Cooling power of No I dilution refrigerator

A - 0.21 mW still power

B - 0.86 mW still power

C - 2.56 mW still power

D - 5.40 mW still power



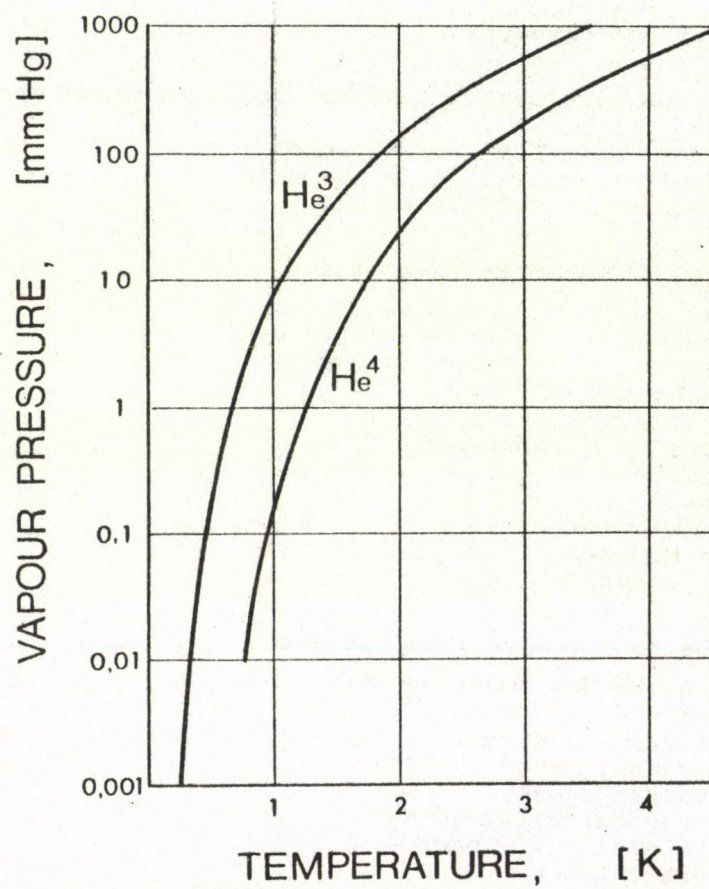


FIG. 1



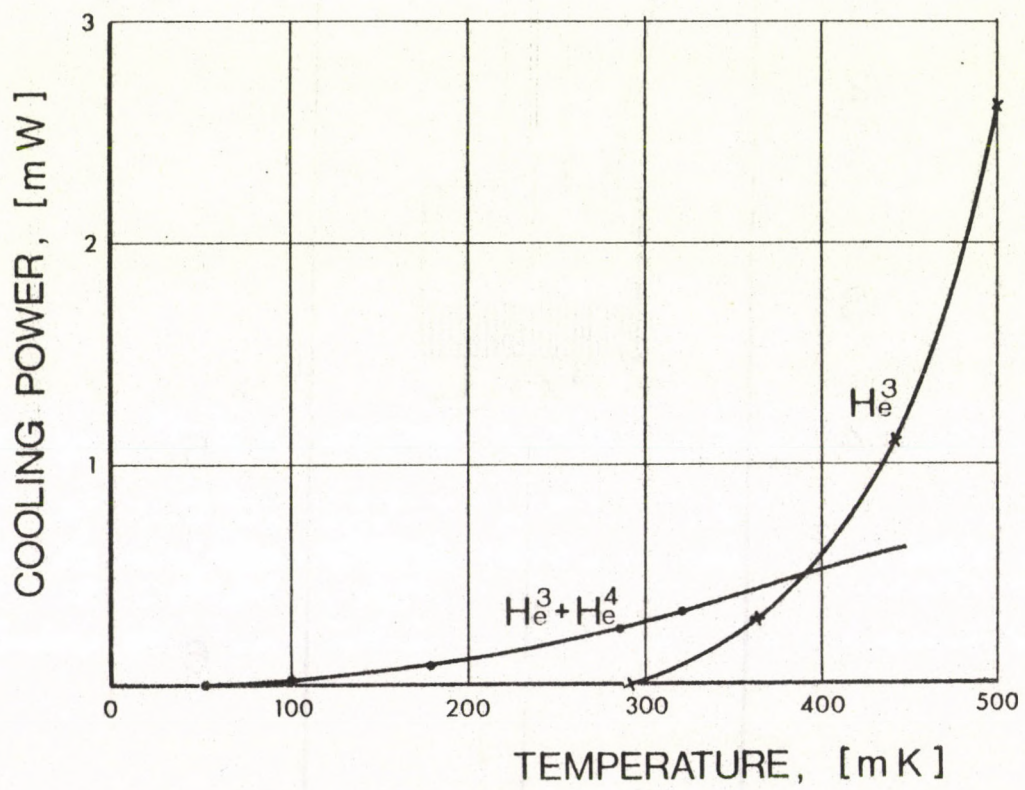


FIG. 2



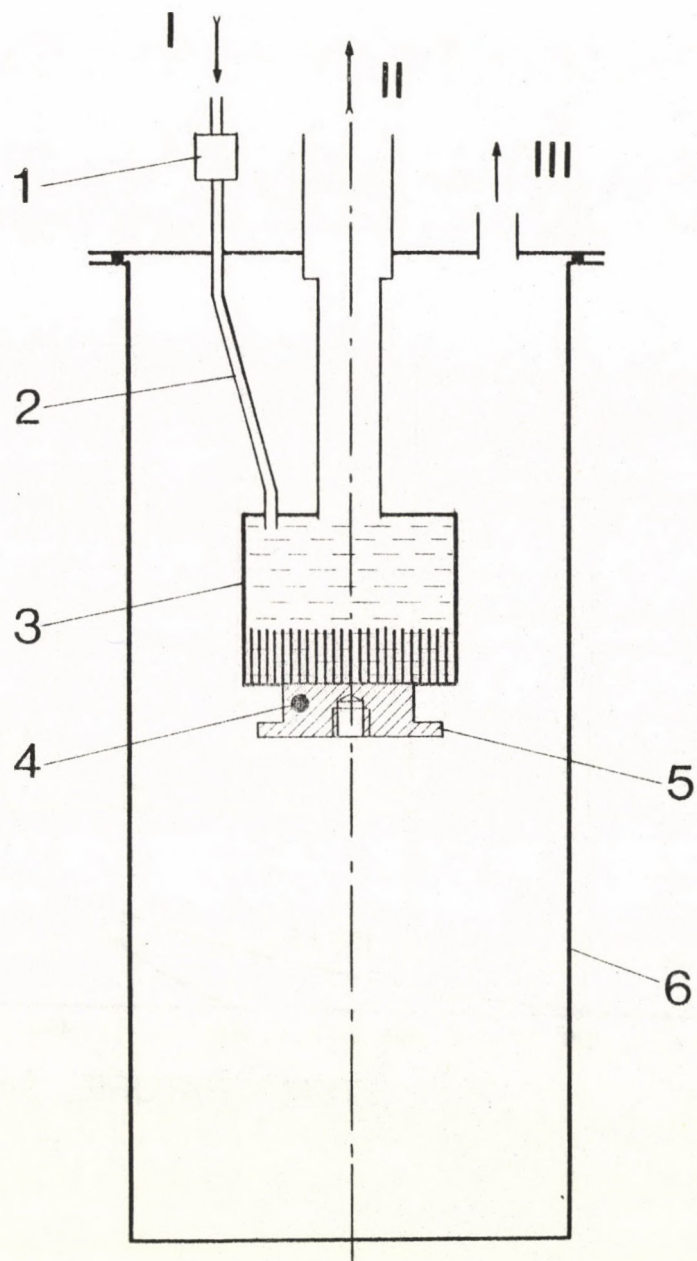


FIG. 3



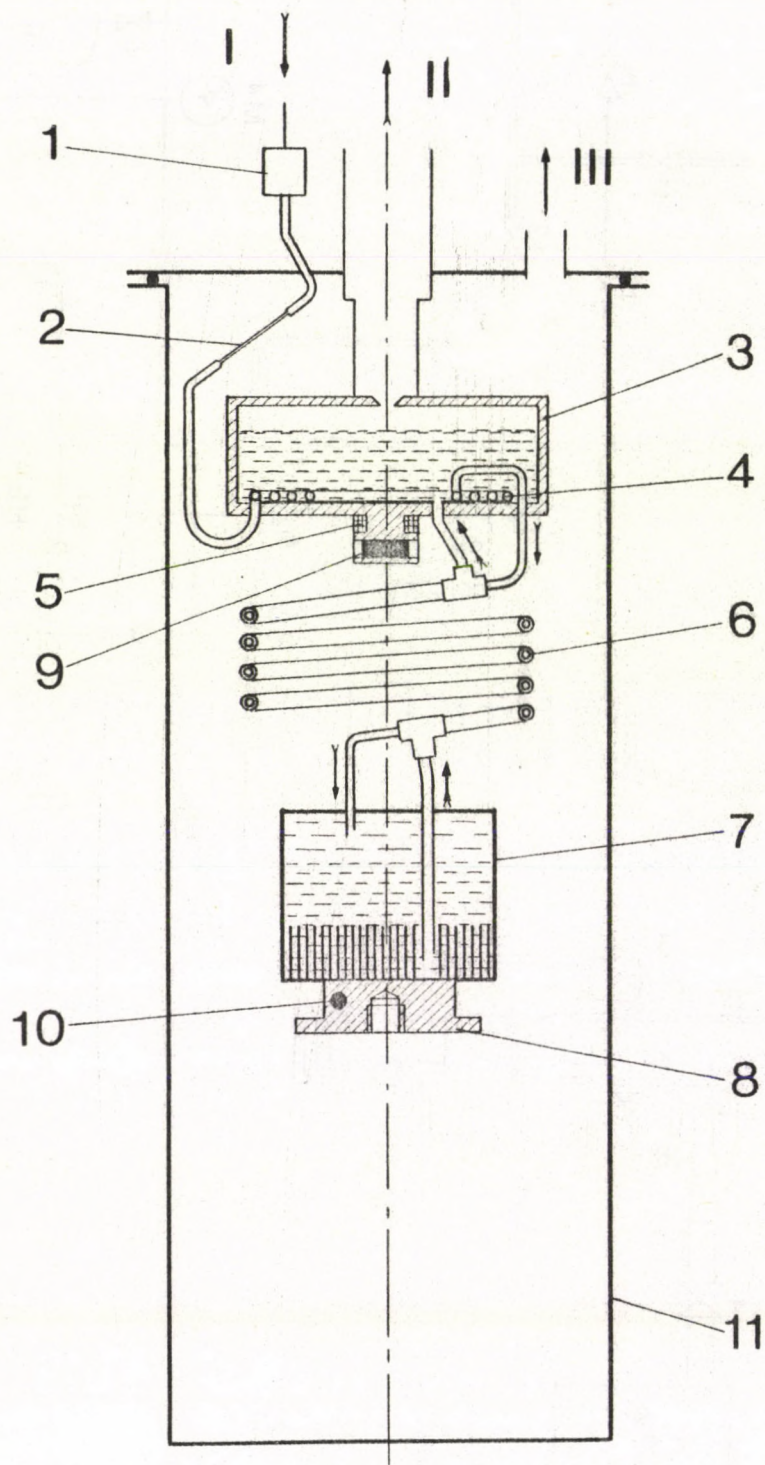


FIG. 4



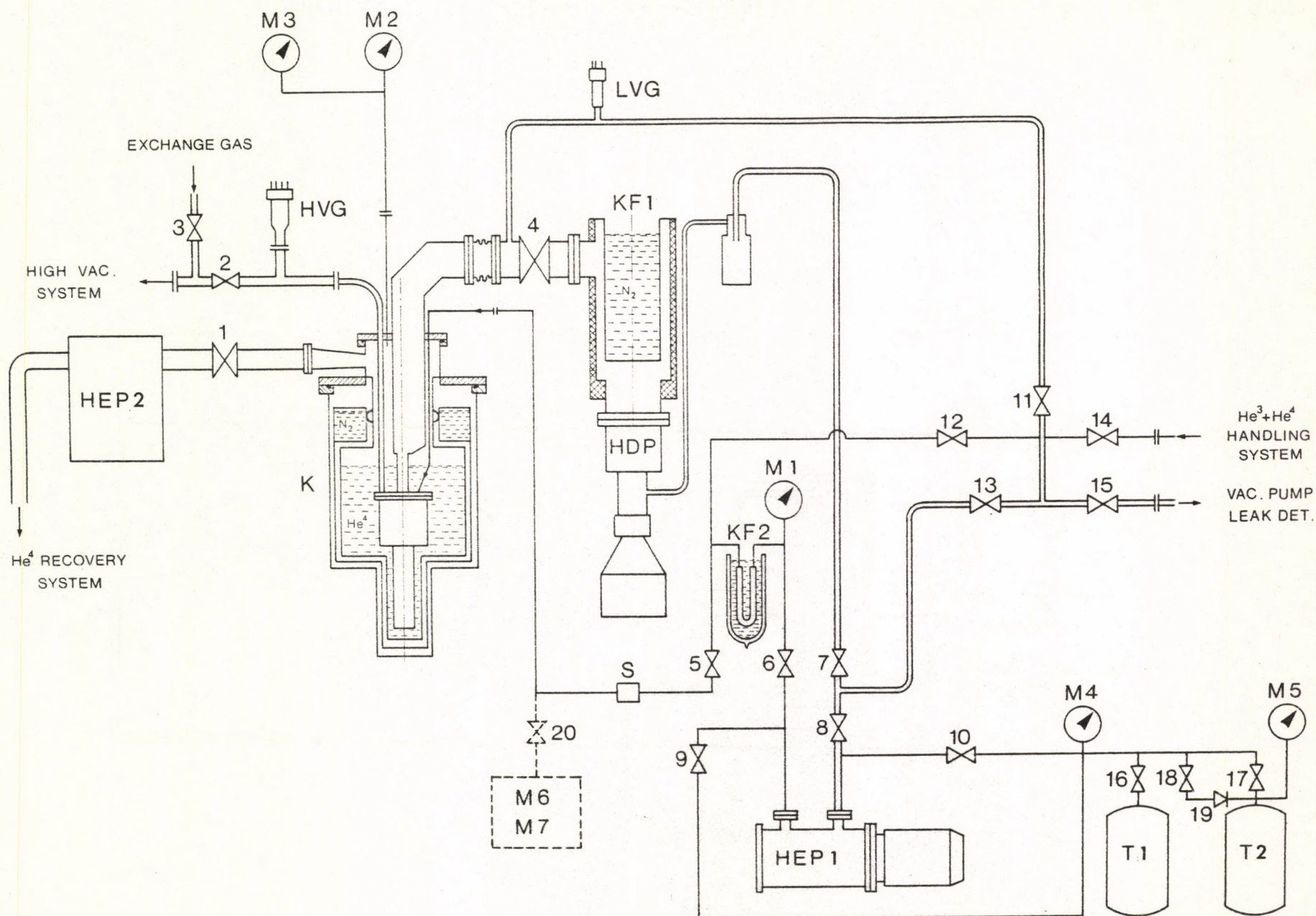


FIG. 5



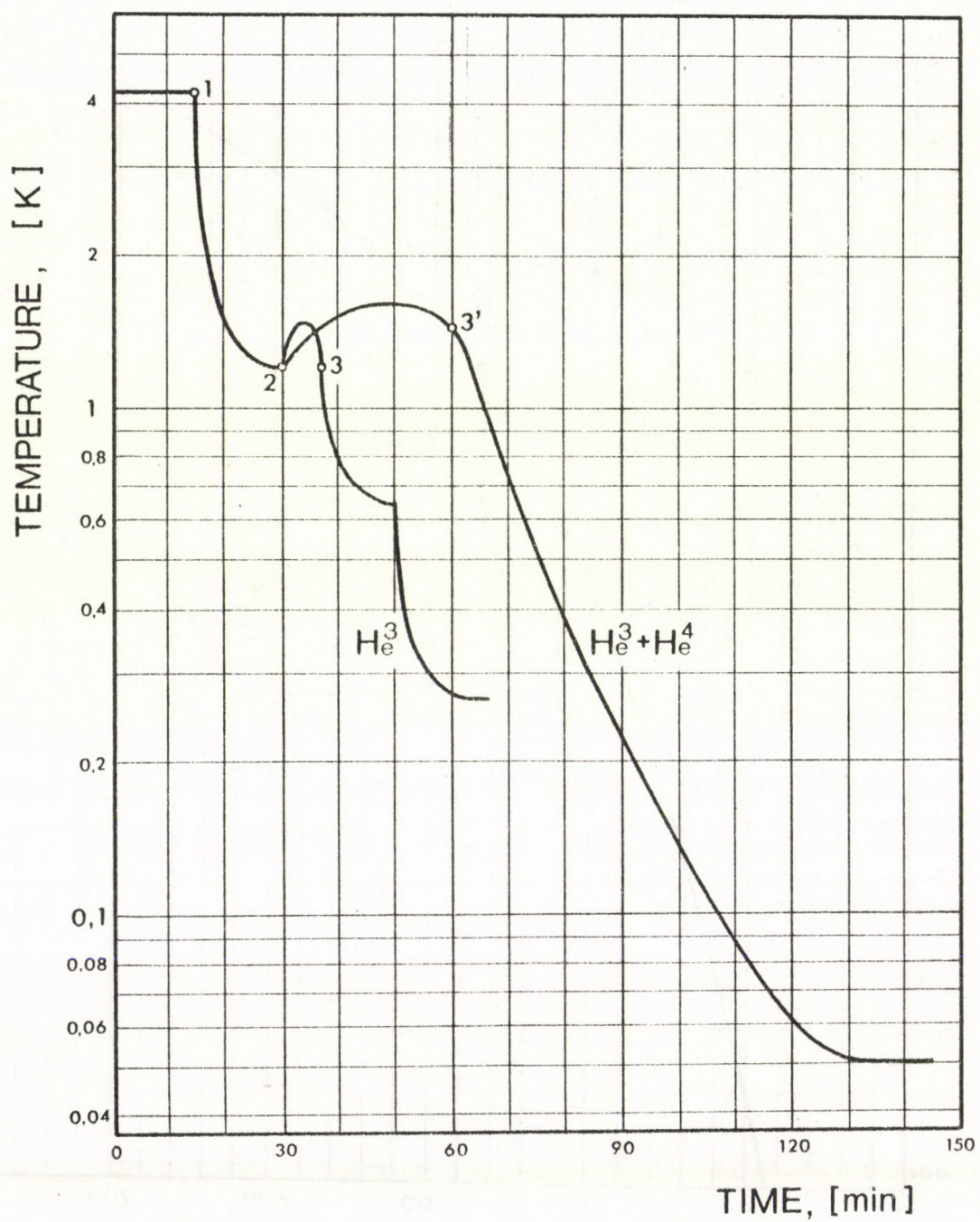


FIG. 6



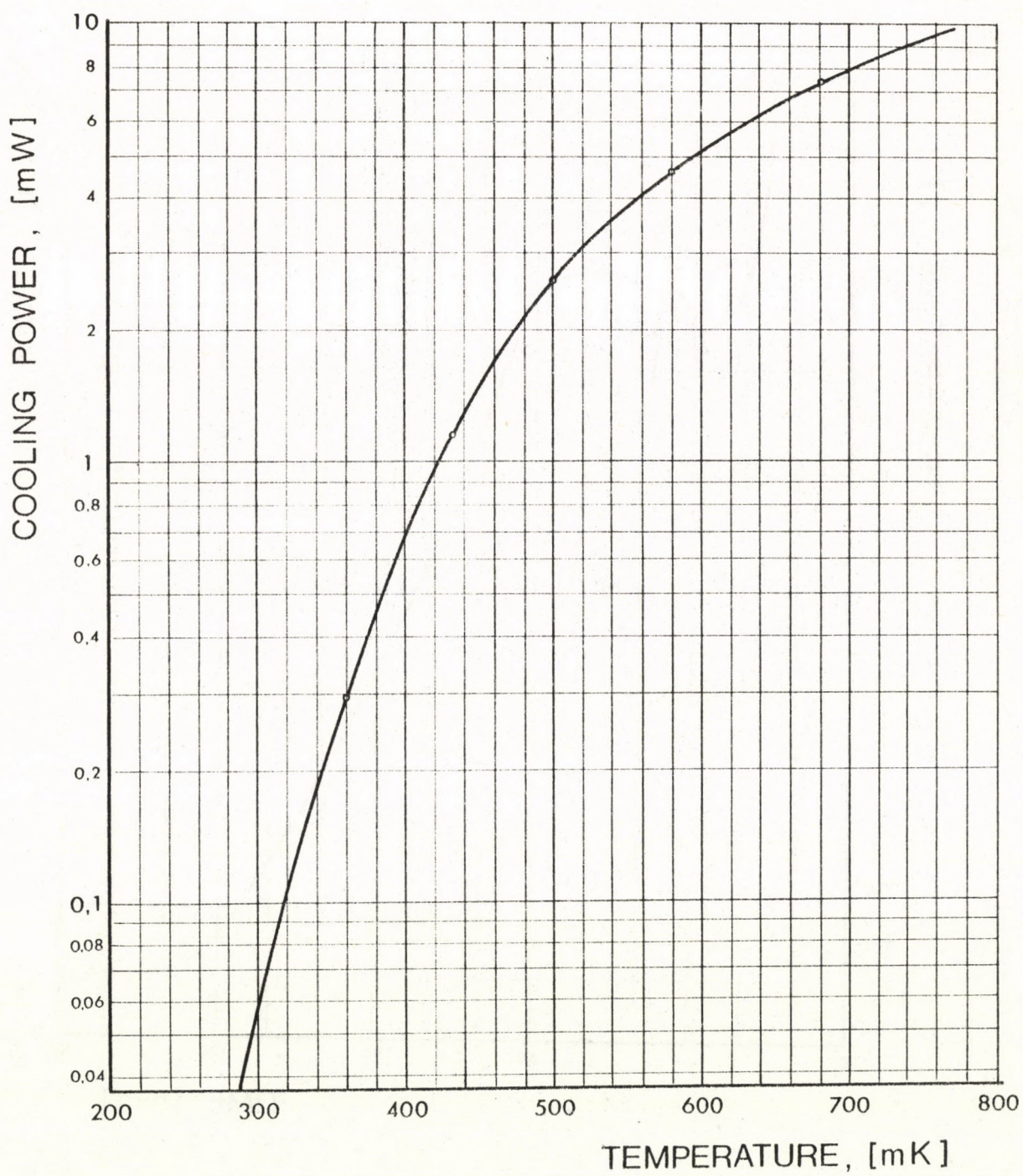


FIG. 7



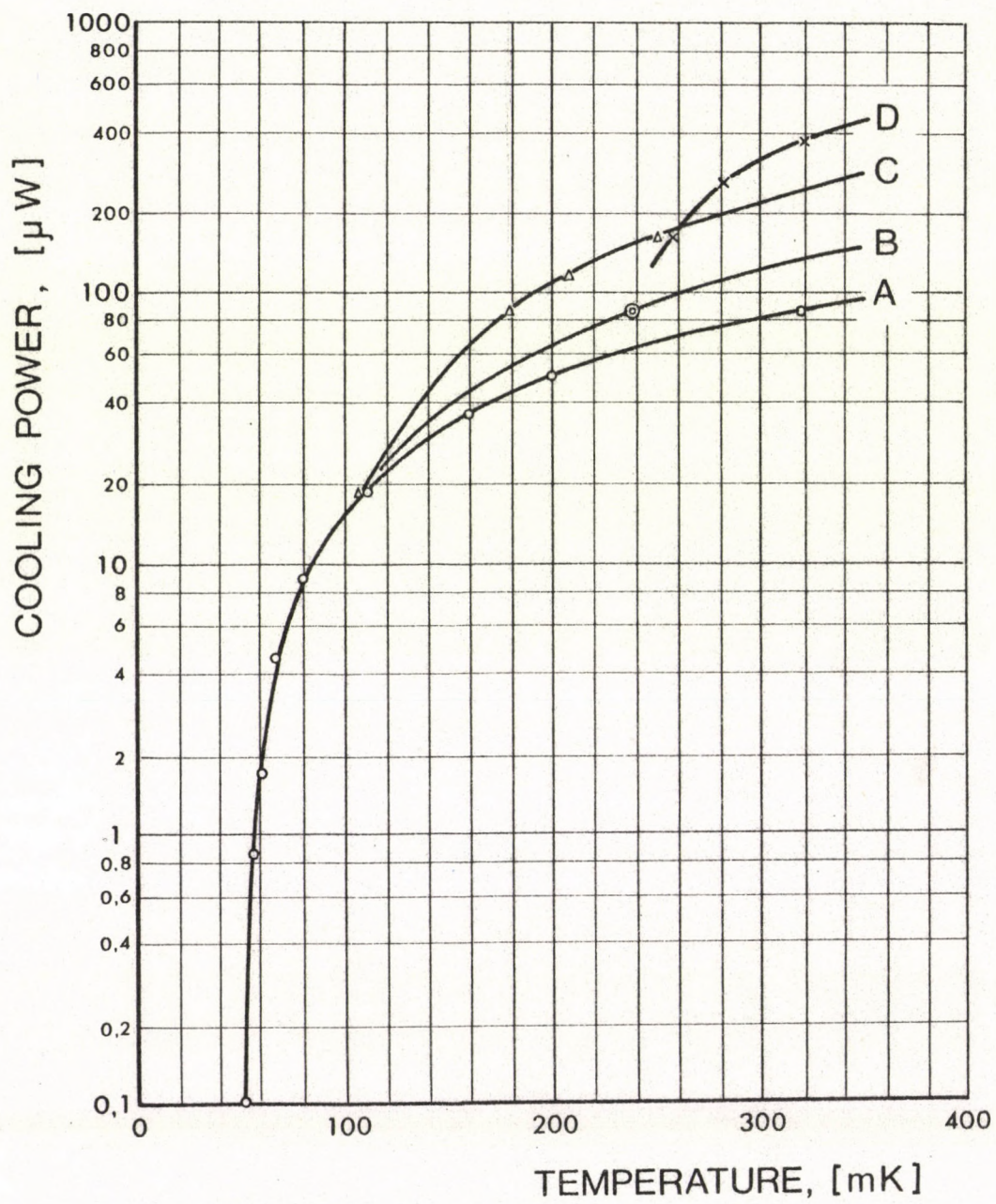


FIG. 8





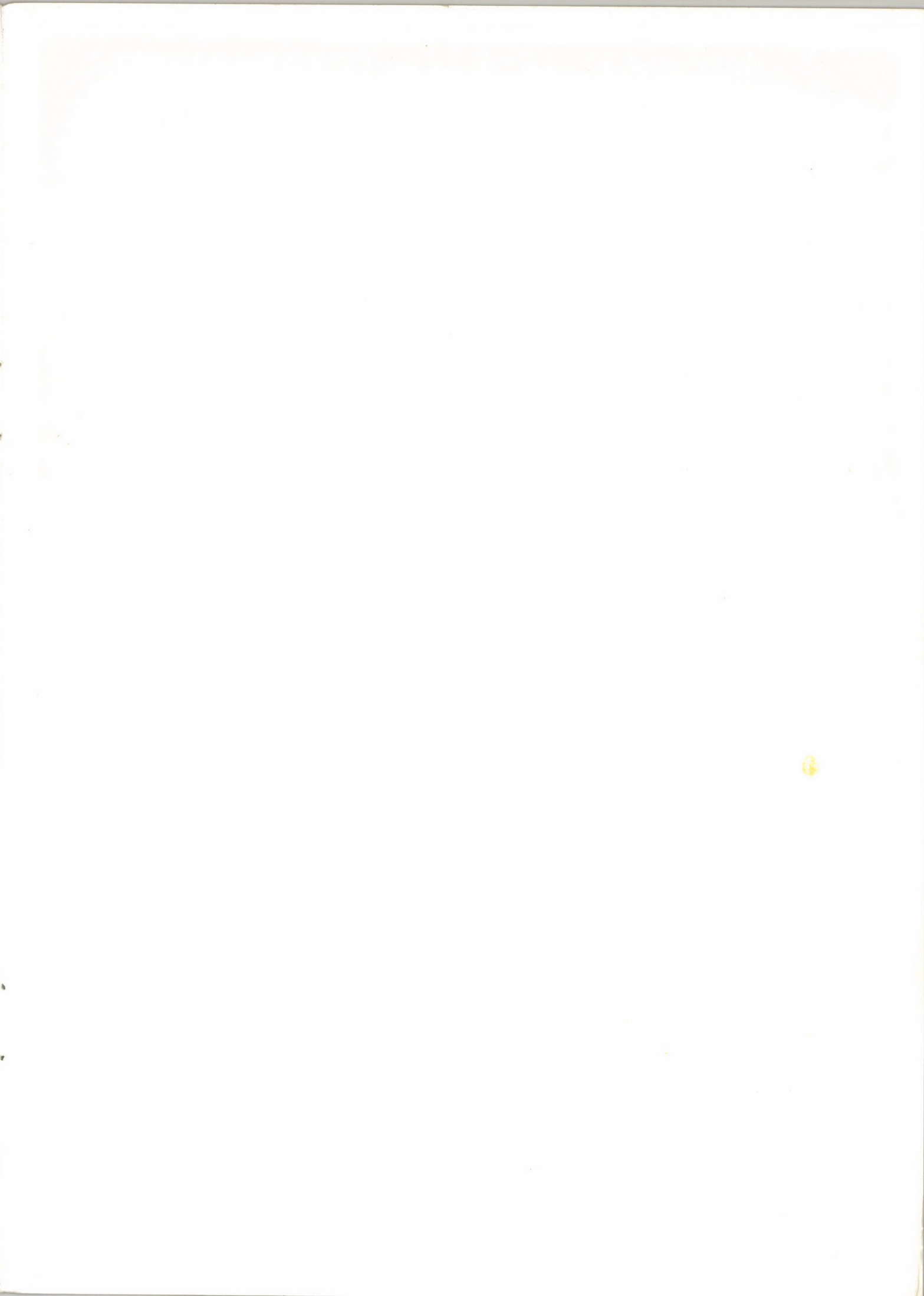














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